

THERMAL PROCESSING OF LOW-GRADE GLYCEROL TO ALCOHOLS FOR BIODIESEL PRODUCTION

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**National Institute for Advanced Transportation Technology
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16. Abstract Crude glycerol is proposed to be converted thermochemically to methanol, ethanol and/or propanols. Such alcohols, or mix of these alcohols, can be re-used in the production of biodiesel to enhance its value. Among the thermochemical conversion processes, liquefaction is preferred since it favors converting organic compounds to liquid products, thus employed in this project. The activities planned for Year 1 were successfully conducted as scheduled, and the initial findings showed promising results. A high temperature, high pressure reactor system (consisting of a 300-mL bench-top reactor) was designed and developed. An enclosed chamber with ventilation and CO alarms was constructed to host the reactor system. Preliminary testing was conducted that helped refine the procedures and control mechanism. Analytical methods for measuring the feedstock and product mixtures were established using GC and HPLC. Based on literature research and analysis to the system, the major process parameters, (i.e. reaction temperature, type and initial pressure of the reducing agents, and time of reaction) were identified and evaluated preliminarily through experiments of batch and fed-batch. Preliminary results showed that temperature and time of reaction significantly affect the conversion of glycerol. The results provided insights on how to continue in Year 2. Further studies will focus on collecting data and evaluating the effects of all process parameters thoroughly. Analytically, further research is still needed to find an alternative solvent for effectively measuring mixtures of methanol, ethanol, propanol and glycerol.			
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1 EXECUTIVE SUMMARY

Crude glycerol is the major by product of biodiesel production. Conversion of crude-glycerol from biodiesel production to value added products can broaden its use and ultimately reduce the cost of biodiesel. In this project, crude glycerol is proposed to be converted thermochemically to methanol, ethanol and/or propanols to enhance its value. Such alcohols or mix of alcohols can be re-used in the production of biodiesel from transesterification of vegetable oils. Among the thermochemical conversion processes (e.g., pyrolysis, gasification, and liquefaction), liquefaction is preferred since it favors the conversion of organic compounds to liquid products. Consequently, liquefaction was employed in this project. This report provides a summary of the activities conducted and the results gathered from the Year 1 of the project.

As scheduled in the Year 1 project, a high temperature, high pressure reactor system was designed and constructed. The system has a 300-mL PARR 4560 Mini Bench Top Reactor from PARR Instruments. A metal-framed chamber with ventilation and CO alarms was constructed to host the reactor system. Preliminary tests using water and glycerol showed that the reactor system works well and can be used for conducting the research. Preliminary testing of the system helped refine the procedures and control mechanism.

Analytical methods for measuring the feedstock and product mixtures were developed using Gas Chromatograph (GC) and High-Performance Liquid Chromatography (HPLC). The procedures were refined and modified based on the information gathered from the preliminary tests. The methodology developed successfully determined quantitatively glycerol in samples by HPLC but not methanol, ethanol and propanol. On the other hand, the GC procedure was able to simultaneously detect methanol, ethanol, 1-propanol, 2-propanol and glycerol in the samples. Comparison of Standard Deviation (SD) values suggests that the results by GC deviated less than those by HPLC.

After literature research and analysis to the system, major process parameters were identified and evaluated by preliminary experiments. Reaction temperature, type and initial pressure of the reducing agents and time of reaction were identified as the major process parameters. Experiments of batch and fed-batch were able to evaluate the effects of these major process parameters over a wide range of conditions. A feeding system was designed and equipped to the reactor system to conduct fed-batch experiments. Results showed that temperature and time of reaction significantly affect the thermochemical conversion of glycerol. Moreover, it also suggests that an initial CO pressure of 200 psig was too high although this inference is not yet conclusive.

The activities planned for Year 1 were successfully conducted as scheduled and the initial findings showed promising results and provided insights on how to continue in Year 2. Further studies will be done to collect sufficient data and to evaluate the effects of all process parameters thoroughly. Analytically, it was also found that the solubility of glycerol in diethyl ether-pyridine was limited. Further research is still needed to find an alternative solvent for effectively measuring mixtures of methanol, ethanol, propanol and glycerol.

2 INTRODUCTION

The economic competitiveness of biodiesel is significantly affected by the disposal and utilization of its major by product, crude-glycerol. With the production of biodiesel increasing, the supply of glycerol will increase, decreasing its value. Thus, the conversion of glycerol to other value added products can broaden its use, increase its demand and ultimately reduce the cost of biodiesel production.

Thermochemical conversion of glycerol to primary alcohols is a potential process to increase the value of crude glycerol. Thermochemical conversion uses heat and applies chemical reagents to breakdown crude glycerol into simpler compounds. Primary alcohols are organic compounds that have a hydroxyl group at one end of the carbon chain. Glycerol is a three carbon compound with each carbon containing a hydroxyl group. Breaking the carbon-to-carbon bonds or removing some of the hydroxyl groups in the glycerol structure will produce primary alcohols. Methanol, the simplest primary alcohol produced from glycerol, can be used as reactant in the production of biodiesel from triglycerides. Other alcohols, such as ethanol, propanol, and *iso*-propanol, can also be used as an alternative reactant to methanol in the production of biodiesel. Thus, finding ways of producing primary alcohols from glycerol is important.

There are three general thermochemical processes, namely, gasification, pyrolysis and liquefaction. Among these processes, pyrolysis and liquefaction produce relatively high percentage of hydrogenated oils, which methanol and ethanol may be found. Both processes use heat and oxygen-absent conditions. Pyrolysis operates at atmospheric pressure and relatively higher temperatures (400 to 600°C) while liquefaction operates on relatively moderated temperatures (300 to 400 °C) and higher pressures (720-2,900 psi). Moreover, liquefaction favors the conversion of organic compounds to liquid products and minimizes the production of char compared to pyrolysis. With this, liquefaction is preferred in producing liquid products.

An applied research entitled “Thermal Processing of Low-grade Glycerol to Alcohols for Biodiesel Production” was proposed and supported by NIATT. This research project aims to conduct a process investigation on glycerol thermochemical conversion, and an engineering evaluation on the technology. The project consists of two phases and is expected to be finished by December 31, 2009. Once successful, the project will contribute to the current knowledge base on the utilization of crude glycerol and eventually benefit the whole biodiesel industry.

3 OBJECTIVES

The purpose of this report is to provide a summary of the activities conducted and the results gathered from the Year 1 of the project. Specifically, the objectives of the Year 1 of the project are the following:

1. To develop and test a reactor system that is capable of conducting high temperature, high pressure chemical reaction of thermochemical conversions,
2. To build a chamber that will harness the pressure reactor,
3. To develop analytical procedures for detecting and identifying the products from the thermochemical conversion of glycerol,
4. To identify major parameters that affect the thermochemical conversion of glycerol to alcohols, and
5. To develop the experimental design for the Phase II of the project.

4 ACTIVITIES CONDUCTED IN YEAR 1 PROJECT

4.1 Development of the Reactor System

A 300-mL PARR 4560 Mini Bench Top Reactor was purchased from Parr Instruments and used for this research (Figure 1). This reactor can handle up to 3,000 psig of pressure and 350 °C of temperature. It is controlled by the 4857 Reactor Controller that controls temperature, pressure and motor speed using a dual thermocouple, pressure transducer and tachometer, respectively. For safety measures, a metal-framed chamber was constructed to enclose the reactor and exhaust out in case of gas leakage or unexpected burst of the working media occurs during the experiment. A schematic drawing illustrating the gas and cooling water piping and cables for the control is shown in Figure 2. Controls of the main valves, switches and the control console, PARR 4857 Reactor Controller, are placed outside the chamber for easy access. Figure 3 shows the metal-framed chamber with the connections and gas tanks. The copper pipes deliver cooling water to the reactor and direct excess gases to the exhaust. Carbon monoxide (CO) is one of the reducing agents used in this research. For safety purposes, two CO alarms were installed to notify researchers if a CO leakage occurred and if the CO levels in the surroundings became toxic. One alarm was placed inside the chamber and the other was installed outside.

4.2 Testing of the Reactor System

Preliminary tests using water were conducted to identify any problems such as leaks. The tests showed no immediate problems for the reactor system to be used for the research. The tests also provided information to refine the procedures in operating the reactor system. Operating procedures for the thermochemical conversion of glycerol to alcohols were developed.

Preliminary runs using glycerol were performed using the developed procedures. Results showed that the average heating rates were roughly 10°C/min which is usual for liquefaction (National Science Foundation, 2008). It also showed that the reactor system can sustain the reactor's temperature within the 2 °C range.



Figure 1. PARR 4560 mini bench top reactor.

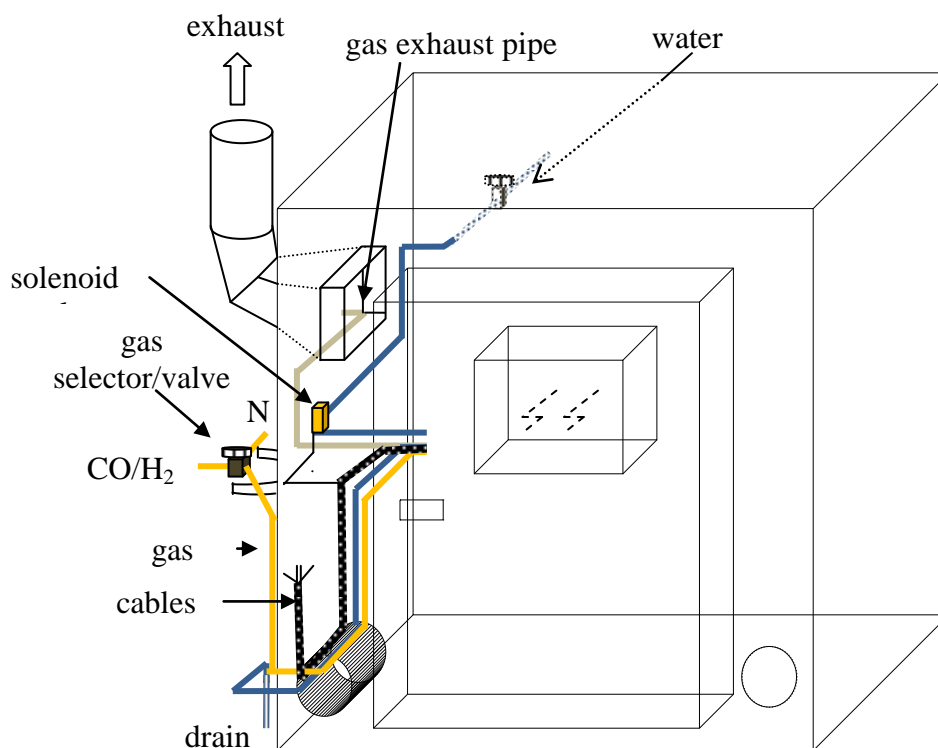


Figure 2. Schematic diagram of the reactor's chamber.



Figure 3. The constructed metal-framed chamber.

4.3 Development and Evaluation of Analytical Procedures

Appropriate analytical methods are necessary to determine the concentrations of glycerol and alcohols in the product after reaction. The physical and chemical properties between methanol/ ethanol/ propanols and glycerol are quite different. While there are established standards and analytical procedures in determining alcohols and glycerol individually, there are no standard procedures for specifically determining methanol, ethanol, propanols and glycerol in their mixtures. Gas chromatography has been used in determining small quantity of glycerol in biodiesels (ASTM, 2007). Thus, gas chromatography could very well be the potential analytical method in measuring the concentrations of the various components of the product mixture necessary in studying the thermochemical conversion of glycerol.

The Biofuels Analytical Laboratory at the Department of Biological Agriculture Engineering (BAE) is equipped with both GC and High-Performance Liquid Chromatography (HPLC). Thus, both were employed for detecting glycerol, methanol, ethanol, 1-propanol and 2-propanol individually and in their mixtures. The information gathered from secondary research provided the starting point in the development of the methodologies for GC and HPLC. Alltech Altima C18 and Ultracarb columns were used in HPLC while DB-wax column was used in GC. Preliminary runs were performed to refine the methodology and procedures.

The methodology developed for HPLC successfully determined glycerol present in samples but not methanol, ethanol and propanols. These compounds were not detected because ELSD, which is the detector used by the HPLC, cannot detect volatile compounds such as methanol. On the other hand, GC uses FID which was able to detect all the compounds present in the sample. Table 1 summarizes the computed relative standard deviations of each compound and determination methods. The RSD values suggest that the results by GC deviated less than those by HPLC. Moreover, the deviations on the responses in determining glycerol, ethanol and methanol did not differ significantly.

Table 1. Relative Standard Deviations of the Compounds through Different Determination Methodologies

Compound of interest	Relative Standard Deviations (percent)	
	HPLC with Alltech Altima C18 column	Gas chromatogram with DB-wax column
Glycerol	4.7199	3.2839
Methanol	---NA---	3.8362
Ethanol	---NA---	3.2159

Another issue in using the HPLC in determining glycerol and other alcohols is that the repeatability shown in Tables 2 and 3. The p-value for the HPLC was less than 0.05. This suggests that results by HPLC significantly varied between replicates while the results by GC did not.

Table 2. ANOVA Results on the Day-to-Day Comparison Using HPLC

Source	DF	Type III SS	Mean Square	F Value	p-value
Replication	2	3.08	1.54	17.79	0.0007
Amount	5	62.18	12.4	143.42	<.0001

Table 3. ANOVA Results on the Day-to-Day Comparison Using Gas Chromatography

Source	DF	Type III SS	Mean Square	F Value	p-value
Replication	1	1.27 E11	1.27 E11	0.44	0.5434
Amount	4	1.43 E14	3.58E13	124.04	0.0002

Figure 4 shows the plot of the computed response area on the gas chromatogram against the amount of glycerol. The trend shows that it curves at more than 600 mg of glycerol. This means that the methodology developed on this specific GC can accurately determine glycerol up to 600 mg. One possible reason for this limitation is the lower solubility of glycerol to diethyl ether-pyridine solution. This limitation was not observed for ethanol, methanol and propanol.

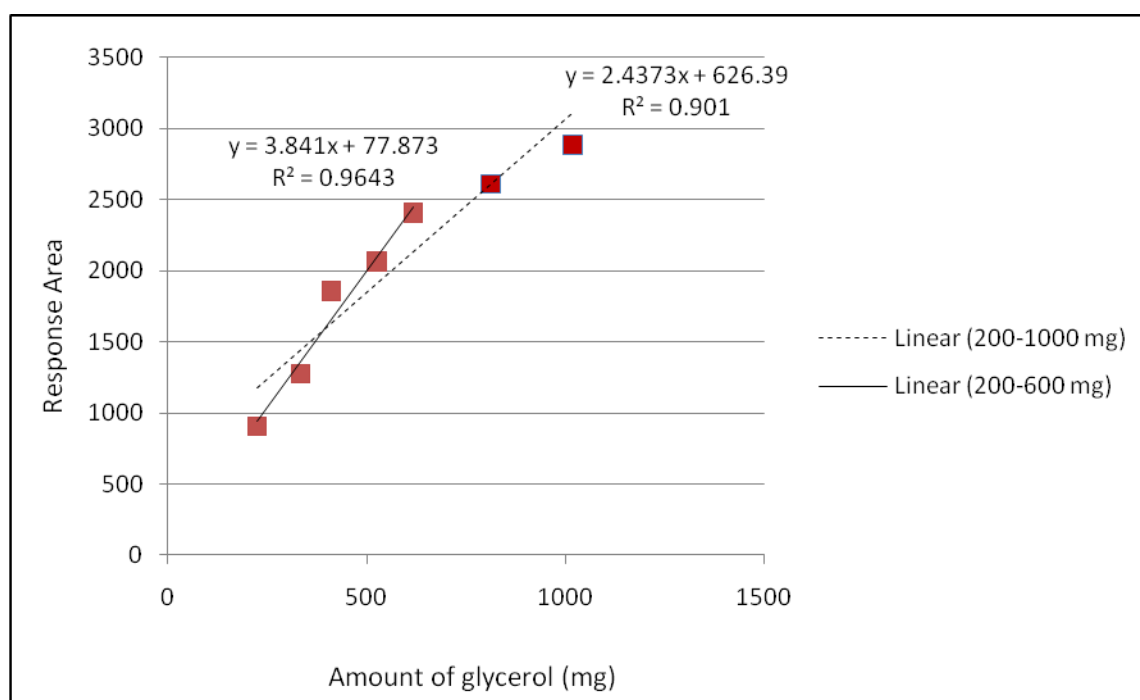


Figure 4. Plot of response area on the gas chromatogram against amount of glycerol.

4.4 Identification and Determination of Major Process Parameters

The major process parameters were preliminarily identified based upon knowledge and literature research. They were then evaluated through test runs.

4.4.1 Identification of Major Process Parameters

Reaction time and temperature, initial pressure of the reducing agent, and the type of reducing agent were initially identified as major process parameters for the thermochemical conversion of glycerol. In thermochemical conversion, heat is provided to break down the chemical bonds of a compound and produce free radicals that will eventually attack other molecules to form simpler ones. Temperature of the reaction plays an important role in converting chemicals like glycerol. As the temperature increases, more energy is made available for the reaction. Studies reported that at atmospheric pressure, glycerol undergoes thermochemical conversions at temperatures higher than 430°C (Hurd, 1929). However, no studies were found regarding the effects of the combination of temperature at elevated pressures and with a reducing agent.

As the pressure indicates how much mass of the reducing compound is present in a specified volume, the initial pressure of the reducing agent could play a major role in the thermal conversion of glycerol. Higher initial pressure means more reducing agent is available for the reaction. In addition to the pressure, type of reducing agent, and the time of reaction are other important factors in the process. However, their effects on the thermochemical conversion of glycerol have yet to be explored.

4.4.2 Evaluation of the Process Parameters

One of the objectives of the Year 1 research was to conduct preliminary experiments to explore the range of reaction conditions in order to evaluate the major process parameters. Therefore, experiments were designed to investigate the effects of temperature, initial CO pressure, reaction time and type of reducing agent on the thermochemical conversion of glycerol. The first experiment was intended to determine whether thermochemical conversion of glycerol occurs similar to what happens during fast pyrolysis. Three parameters were studied with a 2³ factorial experimental design as summarized in Table 1. The levels of temperature used in the experiment were 280°C and 330°C which are the claimed thermal decomposition temperature of glycerol and the near the maximum temperature limit that the reactor can operate, respectively. The initial CO pressures, which reflect the initial number of moles of the reducing agent, were set at 50 and 200 psig.

Table 4. Factorial Experimental Design Matrix in Studying the Thermo-Chemical Conversion of Glycerol

Treatment	Temperature (°C)	CO/ initial pressure (psig)	Reaction time (min)
1	330	200	5
2	330	200	1
3	330	50	5
4	330	50	1
5	280	200	5
6	280	200	1
7	280	50	5
8	280	50	1

A fed-batch reactor system was also designed and used to determine whether repolymerization occurred during the experiment. Repolymerization is a process where a compound breaks down into fragments at lower temperature and forms polymerized compounds (Whitehurst, 1938). These polymerized compounds do not decompose at higher temperatures. This prevents the production of desired liquid products. The fed-batch reactor system has an inlet system that was connected to the reactor's sampling port. The inlet system was composed of Eldrex pump which can deliver 30 mL/min of water and can operate up to 3,000 psig. To overcome the high viscosity of glycerol at ambient temperature, glycerol was preheated to 70 °C in order to maintain its flow rate into the reactor. The schematic drawing of the reactor's preheater-mass scale set up is illustrated in Figure 5. The procedure used in the fed-batch experiment was similar to the first experiment.

A third set of runs used a fractional factorial experimental design as shown in Table 2. This design was chosen because it is appropriate for conducting preliminary screening of parameters. This set used similar temperature settings as that of the first set while initial CO pressures were set at 200 and 340 psig. Moreover, time of reaction was prolonged to 3 and 6 hours. This set of runs was designed to determine whether thermochemical conversion of glycerol occurs slowly at longer reaction time. Lastly, to have information on the effect of the type of reducing agent, glycerol was heated to 330 °C at 340 psig of hydrogen and reacted for 3 hours.

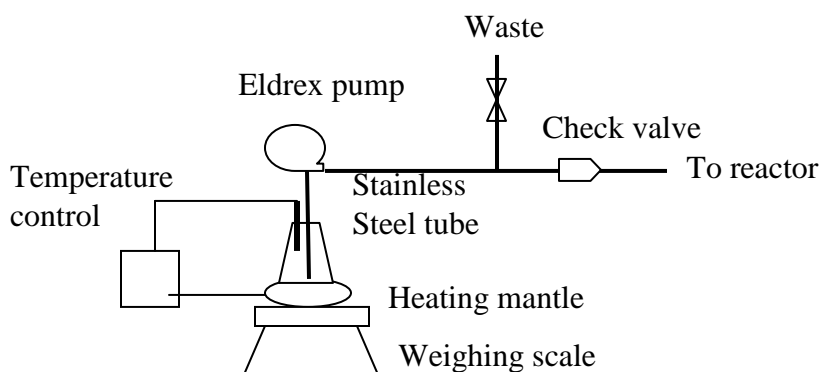


Figure 5. Schematic Drawing of the Inlet System of the Fed-batch Reactor.

Table 5. Fractional Factorial Experimental Design Matrix in Studying the Thermo-Chemical Conversion of Glycerol at Longer Time of Reaction

Treatment	Temperature (°C)	CO/ initial pressure (psig)	Reaction time (min)
9	280	200	180
10	330	340	180
11	330	200	360
12	280	340	360

4.4.2.1 Effects of reaction time and temperature

The thermochemical conversion of glycerol did not occur within 5 minutes of reaction at both 280 and 330°C and 50 and 200 psig initial CO pressures. In 5 minutes of reaction time, glycerol remained colorless and viscous in all the treatments (1-8) as shown in Figure 6. Moreover, bio-oil and char were not seen. On the contrary, all liquid samples collected after 3 and 6 hours (Treatments 9-12) were yellowish to brownish (Figure 7). The color of the samples can be attributed to the presence of other chemicals formed during the reaction. Char and bio-oil were produced in Treatments 10 and 11. On average, 47 percent by weight was bio-oil that had a viscosity similar to water. As expected, the char produced solidified in just a few days. The samples collected from Treatments 9 and 12 did not have char and bio-oil but only a single phase viscous liquid. The viscosity of the samples indicates that most of the glycerol did not undergo thermo-chemical conversion. It should be noted that Treatments 10 and 11 had a higher reaction temperature of 330°C as compared to 280°C in Treatments 9 and 12. The formation of char and bio-oil in the second set of runs, but not in the first set, gives evidence that reaction time is a major factor affecting the thermochemical conversion of glycerol.



Figure 6. Glycerol after one and five minutes of reaction in different treatments.



Figure 7. Glycerol after three and six hours of reaction in different treatments.

4.4.2.2 Effects of initial pressure of the reducing agent

Statistical analysis was done by estimating the Lenth's pseudo-standard error using Yates procedure. This estimated error was used to estimate the t-statistic values of each treatment. It,

did not, however, give enough evidence that the effect of the initial pressure of carbon monoxide is significant. This may suggest that CO is in excess for treatments 9 to 12.

Table 6. Results of T-Tests Using Lenth's Pseudo-Standard Error

Source	T-statistic	T _{critical} (t _{0.05,1})	Result of the test
BC = A	6.980955	6.314	Reject
AC = B	0.837281	6.314	Failed to reject
AB = C	0.496052	6.314	Failed to reject

Legend: A = Effect of temperature effect; B= effect of CO Pressure; C = effect of time

4.4.2.3 Effects of type of reducing agent

When hydrogen was used as the reducing agent in the thermochemical conversion of glycerol, similar results to those of using carbon monoxide were observed. The sample collected also contained char and bio-oil. The bio-oil was approximately 45.79 percent by weight. The color of the bio-oil was much darker compared to the bio-oil collected from treatments 10 and 11 as shown in Figure 8. It is not yet known what causes the discrepancies in color. Based on these results, using carbon monoxide or hydrogen as the reducing agent did not significantly affect the thermochemical conversion of glycerol. However, further studies are still needed to have substantial evidence for this inference.

4.4.3 Fed-batch reactor system

The results in the fed-batch experiments were similar to the first batch of experiments. The reaction did not produce bio-oil and char as expected. However, unidentified peaks were observed in GC analysis at residence time of 11-12 minutes. These unidentified peaks may have a boiling point higher than those of alcohols and pyridine (115.2 °C). According to the literature, glycerol completely decomposes to acetol, acrolein and to other aldehydes at 450 °C (Hurd, 1929). Acrolein boils at 53 °C and its peak should show earlier than the solvent pyridine. Acetol, on the other hand, with a boiling point of 145 °C, is probably a cause of the peaks. However, further research is still needed to identify these unknown peaks. It was also noticed during the experiments that the flow of glycerol in the inlet system was slow. Viscosity and high pressure of the reactor may have influenced the pumping system.

5 SUMMARY

The research activities in the Year 1 project were conducted as scheduled. Preliminary testing using water and glycerol showed that the reactor system is adequate for conducting the research. Initial pressure of the reducing agent, reaction temperature, type of reducing agent and time of reaction were identified as the most influential parameters for the thermochemical conversion of glycerol to simple alcohols. This was supported by the observation during the experiments which showed that glycerol was converted into bio-oil and char with the presence of carbon monoxide or hydrogen at 330°C and 3 to 6 hours of reaction. Statistical analysis confirmed that temperature and time of reaction significantly affected the thermochemical conversion of glycerol. However, the effect of initial pressure of reducing agents is to be further investigated.

The methodology developed for determining ethanol, methanol, propanol and glycerol using gas chromatography was successful in detecting all the target compounds. Statistical analysis also showed satisfactory repeatability. Thus, the methodology can be used to simultaneously detect ethanol, methanol, propanol and glycerol in samples.

6 RECOMMENDATIONS AND WORK FOR YEAR 2 PROJECT

The results of the experiments provided information that temperature and time of reaction affect the thermochemical conversion of glycerol significantly. However, further studies are needed to sufficiently evaluate the effects of initial pressure and the best type of reducing agents for the reaction. Thus, research will continue with thorough investigations on the effects of the major process parameters in Year 2 of the project. Moreover, the investigation procedure will take into consideration that methanol, ethanol, and propanol were not detected in significant quantity in preliminary experiments. The possible reasons may include (1) the reactions did not go to the pathway as expected under the working conditions, (2) the alcohols produced may further decompose into other compounds, and (3) the analytical procedures may need to be further evaluated to effectively detect the targeted products.

It was also found that the solubility of glycerol in the diethyl ether-pyridine solution is limited. Therefore, further research is needed to find an alternative solvent for mixtures of methanol, ethanol, propanol and glycerol. A possible option is acetone-water solution. The addition of water to acetone increases the solubility of glycerol and other organic-soluble compounds to acetone. This will allow the procedure to detect alcohols of low concentrations. On the other hand, acetone can minimize water to back-flash in the inlet port of the gas chromatograph.

An investigation on effect of metal catalysts is likewise recommended to be added in the future plan of the research project. Metal catalysts have been found to affect the production of allyl alcohols from glycerol and the hydrogenation process in producing alcohols, favoring the production of alcohols instead of other compounds in the thermochemical conversion of glycerol.

The activities for Phase II of the project will keep these in mind and substantiate the conclusions made in Phase I. Activities and experimental designs for the additional tasks for Phase II have been discussed and are under consideration for implementation.

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